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RADIOCHEMICAL DETERMINATION OF URANIUM IN ENVIRONMENTAL MEDIA BY ELECTRODEPOSITION

Harry Levine, et al

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RADIOCHEMICAL DETERMINATION OF URANIUM IN ENVIRONMENTAL MEDIA BY ELECTRODEPOSITION

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by

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PREFACE

As part of its program for evaluating the many aspects of radiation exposure, the Public Health Service maintains a continuing interest in the research and development of analytical methods for uranium concentrations in various media. These methods are useful in connection with the assessment of exposure of population groups to ionizing radiation, and also in connection with the development of guides to the formulation of control measures.

Standards for permissible concentrations of uranium in air and water have been promulgated for occupational exposure.* Public health practice, however, takes into account the principle that "controlling the average exposure of a group of individuals to a value below maximum permissible concentrations may not assure that each individual's exposure will be below this limit."**

After evaluating known analytical methods, the Radiochemistry Support Laboratory of the Division of Radiological Health investigated new techniques and modifications applicable to determinations of micro or macro amounts of uranium in environmental media and certain biological materials. Appropriate environmental samples may be air filters, food, water, soil, and vegetation - all of which are collected under PHS radiation surveillance activities.

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* International Atomic Energy Agency: "Basic Safety Standards for Radiation Protection, Safety Series No. 9," Vienna, Austria (1962); and

National Committee on Radiation Protection: "Maximum Permissible Concentrations of Radionuclides in Air and Water for Occupational Exposure," National Bureau of Standards Handbook 69 (June 5, 1959).

** Schultz, N. B., and A. F. Becher: "Correlation of Uranium Alpha Surface Contamination, Airborne Concentrations, and Urinary Excretion Rates," Health Physics 9:901-9 (September 1963).

ABSTRACT

Uranium is isolated by either of two techniques: adsorbing on a strongly basic ion-exchange resin pretreated with 6 N HCl, washing with 9 N HCl to remove interfering ions, and eluting with 1 N HCl; or salting with aluminum nitrate, followed by extracting with ethyl acetate and stripping with water. It is then electrodeposited on a nickel-plated copper disc from a dilute ammonium oxalate solution. Electroplating efficiencies are about 85 to 95 percent. A regulated power source connected in parallel permits several electroplating operations to proceed simultaneously. The procedure has been applied to a series of spiked solutions with reproducible results. Maximum deviation is less than 5 percent for any one determination. Beer's law is followed over a range of 10^{-5} to 5.0 g. uranium. The method is applicable to samples of biological materials as well as to environmental media.

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INTRODUCTION

The analysis of trace quantities of uranium in complex matrices, such as exist in nature and can be present in many environmental media, has been the subject of extensive research and development. At present such analyses are usually carried out by spectrophotometry (11, 14), colorimetry (8, 16), coulometry (5, 16, 20), polarography (13, 21), or fluorometry (6, 11). All these are time consuming and require elaborate chemical processing prior to measurement. The fluorometric procedure is by far the most sensitive and has a detection level of 10^{-8} to 10^{-10} g. of uranium.

This investigation was undertaken to study the feasibility of using an electrodeposition method for the determination of uranium in environmental samples and then counting the alpha activity in a proportional counter. Isolation of the uranium was accomplished by either of two techniques. The first of these is an ion-exchange procedure. The studies of Benerjee and Heyn (3), Banks and others (2), Brody and others (4), and Sporek (18) have demonstrated that ion-exchange techniques are useful for the analysis of traces as well as macro amounts of uranium. Fisher and Kunin (8) employed an anion-exchange procedure to separate uranium from all likely interfering radionuclides, using a sulfate medium; Kraus and others (13) investigated extensively the absorption of uranium from a chloride medium. Several ion-exchange procedures for isolating the uranium prior to electrodeposition were considered, each of which depends on the formation of a complex uranium anion as a chloride (2), or as a sulfate (8).

The sample treatment outlined in this report prepares the uranium in a form conveniently adaptable to the chloride procedure. Iron, the element occurring most commonly with uranium, is adsorbed with uranium on the column, but does not interfere with the detection of uranium as it does in the fluorometric method. The other natural radionuclides which could interfere radiometrically, such as potassium-40, thorium, and radium, are not adsorbed by the column. Whitson and Kwasnowski (20) measured the radioactivity from enriched uranium alpha emissions following an electrodeposition separation. Their method involved the electrodeposition of uranium on nickel and determination of the alpha activity of enriched uranium in urine.

The second method outlined in this report for isolating uranium is based on solvent extraction. The extraction of uranium with ethyl acetate using aluminum nitrate as a salting agent (11) requires little time and the simplest of equipment to isolate uranium from interfering elements. A stripping extraction with water, following the principal extraction, ensures a quantitative separation of uranium prior to electrodeposition.

The electrodeposition procedure used is that of Alercio and others (1), which employs nickel discs. Nickel precedes copper in the electromotive series of the metals; hence, it was decided to plate out nickel on copper from an alkaline nickel solution and use the polished nickel-plated copper discs for the electrodeposition of uranium. Electroplating of nickel on copper discs has several advantages. Where large numbers of determinations are to be made, the savings are appreciable. The cost of nickel is at least four times that of copper. Moreover, copper planchets are available on the market, whereas nickel planchets have to be stamped out by a special milling process to meet the specifications of the user, adding materially to the cost. By plating the nickel out on a clean copper surface before use, a minimum of polishing (important for satisfactory use of the planchets) is required. The nickel solution used for plating can be used repeatedly; as many as 100 samples can be analyzed from approximately 15 g. of nickel sulfate solution. The time involved in plating nickel out on the copper disc is only 7 minutes. Since the planchets cannot be reused after the analysis, the economy of using discardable copper discs rather than nickel discs is substantial.

The electrodeposition unit used has a regulated power supply and is connected in parallel to permit several electroplating operations to proceed simultaneously and independently of each other. The capability of the power supply for optional voltage output permits closer regulation of the electrode potential, thereby resulting in a consistent electroplating efficiency. The line voltage is 115 a.c., and the output 23 volts d.c. The cells are Teflon cylinders, measuring 45 mm. outside-diameter and 31 mm. inside-diameter by 50 mm. in length. Uranium is deposited on nickel-plated copper discs, 45 mm. in diameter and .032 mm. in thickness. Platinum stirring rods adjustable in height and speed are used as anodes and the housing of the electrodeposition unit is used as the cathode.

The essential features of the method are complete decomposition of the sample by classical methods (11, 12), isolation of uranium by either of two techniques described, and electrodeposition of uranium on nickel-plated disc in alkaline solution.

DETAILED PROCEDURE

I. Decomposition of Sample

Depending on the nature of the sample, one of the following methods of decomposition is applicable:

A. Weigh 1.000 g. ashed, finely ground sample in beaker. Add 20 ml. 1:1 HNO_3, digest on hot plate for 1 hour, and filter. Discard residue, evaporate filtrate to dryness, and mix with 7.5% HNO_3. For removal of uranium by ion-exchange resin, evaporate the solution to dryness twice with HCl in glass to convert to chloride and mix with 6 N HCl.

B. Weigh 1.000 g. ashed, finely ground sample in platinum dish. Add 10 ml. 1:1 HNO_3 and 10 ml. HF, cover, and digest for 30 minutes on hot plate. Remove cover and evaporate to dryness. Add 10 ml. 1:1 HNO_3 and evaporate to dryness. Repeat with 10 ml. 1:1 HNO_3 and mix with 7.5% HNO_3. For removal of uranium by ion-exchange resin, evaporate the solution to dryness twice with HCl in glass to convert to chloride and mix with 6 N HCl.

C. Weigh 1.000 g. ashed, finely ground sample and transfer to platinum crucible. Mix with 5 g. Na_2CO_3 and fuse at 950° C. ± 20° in muffle furnace for 30 minutes. Cool, leach with water, and neutralize with HNO_3. Add HNO_3 to make 7.5% HNO_3. Centrifuge; pour off supernatant and save. Transfer acid insoluble to platinum dish, add 10 ml. HF, and destroy silica by evaporating to dryness. Evaporate to dryness twice with 1:1 HNO_3, mix with 7.5% HNO_3, and add to main solution. For removal of uranium by ion-exchange resin, evaporate the main solution to dryness twice with HCl in glass to convert to chloride and mix with 6 N HCl.

The sample is now in proper form to isolate uranium by either of the two techniques.

II. Isolation of Uranium

A. Removal of Uranium by Ion-Exchange Resin

1. Solution from I-A, I-B, or I-C is brought to 200 ml. with 6 N HCl. Any hydrolytic precipitate will clear up at this point.

2. Pass solution through pretreated ion-exchange column at a flow rate not exceeding 2 ml. per minute.

3. Wash column with 200 ml. 9 N HCl at the same flow rate.

4. Elute resin column with 150 ml. 1 N HCl at the same flow rate, collect in 250-ml. beaker and heat to dryness.

5. Moisten residue with 7.5% HNO_3 and take to dryness. Repeat twice.

B. Removal of Uranium by Ethyl Acetate Solvent Extraction

1. Solution from I-A, I-B, or I-C above is made of 20 ml. 7.5% HNO_3. Add 38 g. $Al(NO_3)_3 \cdot 9H_2O$ and warm to dissolve. Solution will clear at this point.

2. Transfer solution to a 60-ml. separatory funnel and rinse beaker twice with 10 ml. ethyl acetate. Add washings to funnel. Shake for 1 minute. Discard aqueous layer.

3. Wash ethyl acetate layer twice with 10 ml. aluminum nitrate-nitric acid solution. Extract combined washings with 10 ml. ethyl acetate and combine layers. Aqueous layer is discarded.

4. Strip ethyl acetate layer twice with 20 ml. water and discard organic layer. Transfer water layers to 250-ml. beaker and heat to remove ethyl acetate vapors.

5. Add 20 ml. 5% $(NH_4)_2CO_3$ solution, digest on steam bath, and cool.

6. Centrifuge solution at 2,500 r.p.m. for 10 minutes. Pour off supernatant into 100-ml. beaker and wash precipitate twice with 1% $(NH_4)_2CO_3$ solution and once with water. Add to main solution and discard precipitate.

7. Acidify solution slowly and evaporate to dryness. Add 5 ml. 7.5% HNO_3 and take to dryness.

III. Electrodeposition of Uranium on Nickel-Plated Disc

A. Add two 10-ml. portions and one 5-ml. portion of .016 M ammonium oxalate to residue from II-A (5) or II-B (7), policing beaker with each portion of ammonium oxalate and transfer solution to Teflon cell containing nickel-plated disc.

B. Clamp cell in position on electrodeposition unit (fig. 1); turn on power and adjust to read 40 mA. with anode platinum stirrers at approximately 200 r.p.m.

C. Electroplate for a minimum of 17 hours, preferably overnight. Remove cell without turning off current. Discard electrolyte and rinse cell with distilled water. Reduce amperage and turn off power.

D. Disassemble cell, remove nickel-plated disc, rinse with water and th with ethyl alcohol, flame to cherry red, cool, and place in appropriate holder for alpha counting in proportional counter.

The efficiency of the counter was taken as 42 percent (2π geometry) and this was confirmed using a National Bureau of Standards calibrated alpha source. The background was generally less than 0.1 count per minute.

REAGENTS AND SPECIAL APPARATUS

All chemicals used are reagent grade.

1. Standard Uranium Solution: A stock solution 1 mg. uranium per ml. was prepared by dissolving 0.1179 g. 99.9% pure U_3O_8 in 7.5 ml. conc. HNO_3 with heat. The solution was diluted to 100 ml. with redistilled water.

2. Ammonium Oxalate Solution: .016 M
Weigh 3.27 g. ammonium oxalate, dissolve in water and dilute to 1 liter with water.

3. Ammonium Carbonate: 5% W/V
Dissolve 5.000 g. solid $(NH_4)_2CO_3$ in water, add 5 ml. NH_4OH, and dilute to 100 ml. with water.

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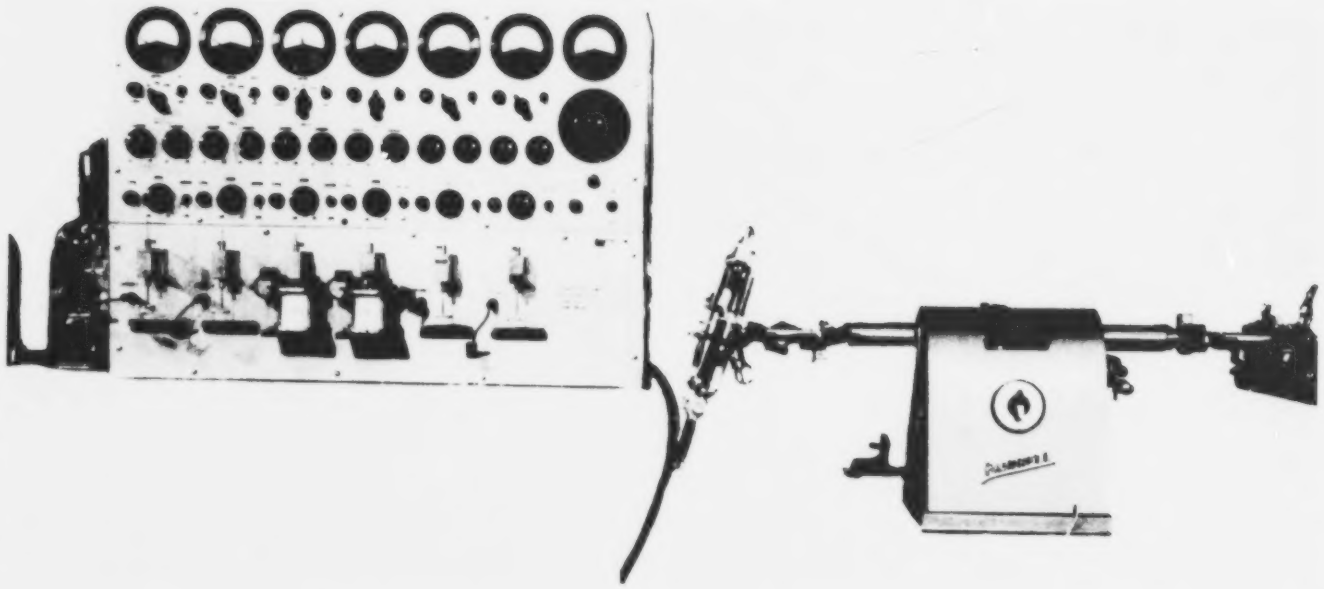

FIGURE 1. Electrodeposition Unit and Shaker

4. Nitric Acid: Conc.; 7.5% V/V

5. Aluminum Nitrate 9-Hydrate

6. Aluminum Nitrate-Nitric Acid Solution:
Dissolve 475 g. $Al(NO_3)_3 \cdot 9H_2O$ in 250 ml. 7% HNO_3. V/V

7. Ethyl Acetate

8. Ethyl Alcohol

9. Hydrochloric Acid: Conc.; 1 N; 6 N; 9 N

10. Nickel Sulfate Plating Solution: 3 M
Dissolve 13.4 g. $NiSO_4 \cdot 6H_2O$ in 100 ml. NH_4OH

11. Preparation of Nickel-Plated Copper Disc

    a. Polish the copper disc to a high luster and assemble in Teflon cell.

    b. Add 10 ml. nickel sulfate plating solution to cell and electroplate at a current flow rate of 150 mA. with anode platinum stirrer at approximately 200 r.p.m. for 7 minutes.

    c. Remove cell without turning off current; then reduce amperage and turn off power. Wash disc with distilled water, dry, and polish to a high luster.

12. Ion-Exchange Column: 10-mm. by 25-cm. column with a high porosity glass filtering disc fused to lower end and fitted with a Teflon needle valve stopper to provide a more reproducible flow rate. A separatory funnel is fitted to the top of the column.

13. Ion-Exchange Resin: Dowex-1-X4 (50-100 mesh) anion resin in a chloride form is used. Fill column with 30 ml. of above resin and wash with 500 ml. 6 N HCl. The column may be used for an indefinite number of separations if it is regenerated after each sample. The flow rate is about 2 ml. per minute.

14. Electrodeposition Unit: The electrodeposition unit built by the electronics section was designed so that at least six cells could be operated simultaneously and independently. The control panel is directly above the cell units for accessibility and convenience. Six current-control sections plus one master voltage-control unit comprise the control panel section. Each cell-control section contains a coarse and vernier current adjustment. A

separate switch and fuse provide individual operation and protection for each unit. A speed-control switch and fuse are also provided for each stirring motor and are at the lower portion of the control panel. This arrangement allows each cell to be operated at a different current density and each anode stirring rod to be driven at a different speed. The anode stirring rods are made of .0625-in. pure platinum wire, inserted into 0.25-in. stainless-steel gear shafts, adjustable in height and locked in place by a thumb screw at the top of each gear case.

The basic circuit in the control panel is a conventional form of regulated direct current. Each cell is connected to the electric power supply in parallel and up to 2 amperes per cell may be used if a high-conductance solution is used in the cell chamber. A 0-50 milliammeter is provided in each cell circuit to indicate current density. A current multiplier selector switch in each circuit provides a current range of 0-50 mA., 0-500 mA., and 0-2 amps. The line voltage is 115 a.c. and the output 23 volts d.c. The cells are Teflon cylinders, measuring 45 mm. outside-diameter and 31 mm. inside-diameter by 50 mm. in length. The copper disc is 45 mm. in diameter and .032 mm. in thickness and is used as the cathode.

15. Centrifuge: Universal model UV (60 cycle, 7.5 a., 115 v.).
16. Shaker: Burrell Wrist-Action, model BB.
17. Internal Proportional Counter: model PC-3A.

EXPERIMENTAL STUDIES

Preliminary experiments were conducted with a standard uranium solution to establish the following:

(a) standard working curve to estimate alpha activity of various concentrations of uranium after electrodeposition,

(b) comparison of two techniques used to isolate the uranium prior to electrodeposition and estimation of the alpha activity recovered,

(c) application of the procedure to a soil sample containing plutonium and spiked with a known amount of uranium to determine interference of plutonium,

(d) application of the procedure to air filters spiked with known amounts of uranium to determine alpha activity recovered,

(e) application of the procedure to water samples spiked with known amounts of uranium to determine alpha activity recovered.

1. A series of synthetic samples containing various amounts of uranium ranging from 2.5×10^{-4} to 5.0×10^{-3} g. of U_3O_8 were processed according to steps III-A to III-D of the detailed procedure. The samples were counted for 60 minutes in the proportional counter (fig. 2). The efficiency of the counter was taken as 42 percent (2π geometry) and this was confirmed using a National Bureau of Standards calibrated alpha source. The background was generally less than 0.1 cpm. Results are shown in table 1 in the appendix. The standard curve is linear and followed Beer's law as shown in figure 3.

2. A series of synthetic samples containing 5 mg. of U_3O_8 was analyzed using both the ethyl acetate extraction and ion-exchange column separation of uranium, to estimate the alpha activity recovered after electrodeposition. Both techniques were analyzed in sextuplicate. The procedure for the ethyl acetate extraction of uranium followed II-B (1-7) of the detailed procedure, except that 5 ml. 7.5% HNO_3 containing 5 mg. uranium standard was extracted with 10 ml. ethyl acetate. The organic layer was stripped with 10 ml. water and the water layer was precipitated with 10 ml. 5% $(NH_4)_2CO_3$ solution to remove the aluminum. The electrodepostion of uranium on the nickel-plated disc was analyzed as described in steps III-A to III-D of the detailed procedure. The procedure for the isolation of uranium by ion-exchange resin followed steps II-A (1-5) and III of the detailed procedure. The samples were counted for 60 minutes in the proportional counter (fig. 2). Results shown in table 2 indicate that the recovery using solvent extraction averaged 78 percent, and the recovery using ion-exchange separation averaged 95 percent.

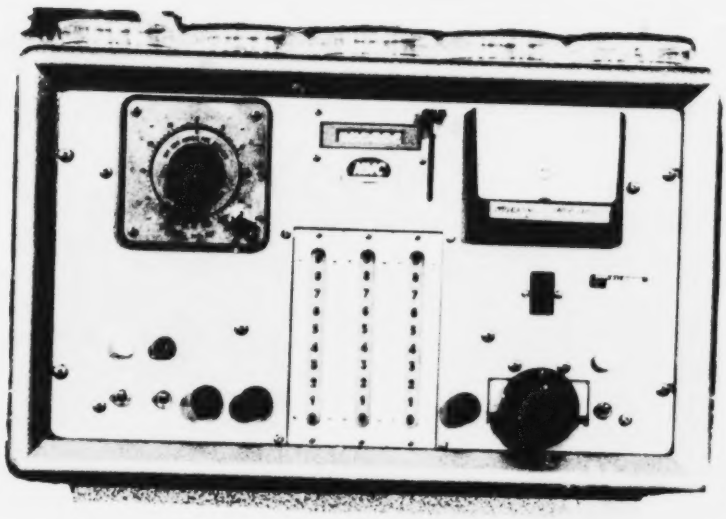

FIGURE 2. Internal Proportional Counter

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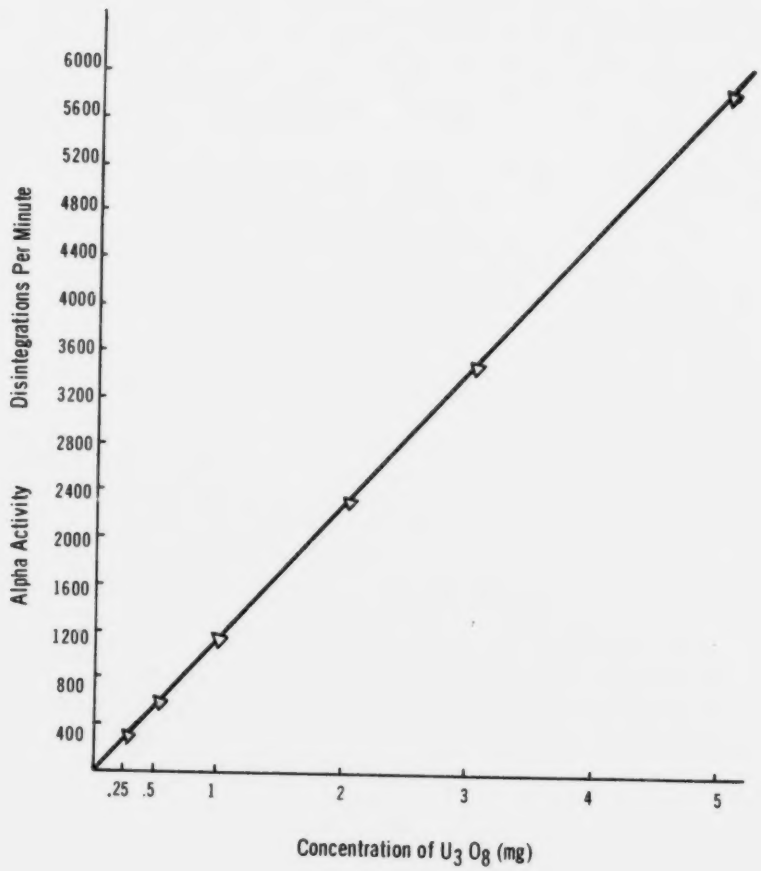

FIGURE 3. Working Curve for Uranium by Electrodeposition

3. Portions of a soil sample representing respectively 0.25 g., 0.50 g., and 1.000 g. of the finely powdered material were spiked independently with 5 mg. of the standard uranium solution and then processed according to I-C, II-A and III of the detailed procedure. The samples were counted for 60 minutes in the proportional counter for alpha activity and interference of plutonium. An analysis of the soil sample for plutonium by electrodeposition (21) showed 181 pc.

   Results are shown in table 3. The recovery for uranium ranged from 80 to 85 percent and plutonium showed no interference. A negative result was obtained on the blank.

4. A number of air filters were spiked independently with 0.1 μg., .05 μg., and .01 μg. of the standard uranium solution and then processed according to I-C, II-A, and III of the detailed procedure. The samples were counted for 60 minutes in the proportional counter (fig. 2). Negative results were obtained on a blank. Results are shown in table 4.

5. Three 500-ml. water samples were spiked with 0.1 μg., .05 μg., and .01 μg. of the standard uranium solution, and coprecipitated with an ammonical alkaline earth phosphate. The precipitate was filtered off, dissolved in 6 HCl, and processed according to II-A and III of the detailed procedure. The samples were counted for 60 minutes in the proportional counter. Negative results were obtained on a blank. Results are shown in table 5.

6. A series of synthetic samples spiked with various activities of a standard uranium solution and ranging from $1x10^{-5}$ to $5x10^{-3}$ g. of U_3O_8 was processed according to II-A and III of the detailed procedure. Beer's law is followed over the range stated above. Results are shown in table 6.

CONCLUSIONS

The series of tests conducted with the procedures described in this paper indicates that, of the two techniques used for isolating uranium for electrodeposition, the method using the ion-exchange resin gives a higher recovery; however, for faster analysis and routine determination of alpha activity, where results could be given quickly in emergency situations, the solvent extraction method is preferable.

A complete analysis of a sample can be accomplished in 3 days. Electroplating efficiencies have ranged consistently from 85 to 95 percent provided that the deposition time, amperage, and alkalinity of the electrolyte are as described. The analytical recovery is also improved by having the discs electropolished. This provides for precision plating of uranium, because a highly polished surface minimizes self-absorption of the alpha count.

A number of specimens were spiked with various amounts of uranium and the percentage recovery determined by comparison with counts obtained from a sample to which the same amount of activity had been delivered directly. Negative results were obtained on all blanks. The mean percentage recovery was 80 to 85 percent with a standard deviation of $\pm$ 10 percent. Samples containing plutonium did not interfere with the uranium recovery. The procedure therefore is effective in separating plutonium from uranium.

The quantity of uranium in the sample can be determined from a plot of disintegrations per minute of standards. The curve is linear and adheres to Beer's law. The limit of detectability is about 7 dpm or 3 pc. of uranium. The method described is recommended on the basis of simplicity, reproducibility, and adequate sensitivity.

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APPENDIX

TABLE 1. Alpha Activity and Percent Recovery for Various Amounts of Uranium

U_3O_8 added (mg.)	Alpha activity found (dpm)	U_3O_8 deposited on disc after electrodeposition (mg.)	Percent recovery
0.25	292	0.22	88.0
0.50	575	0.47	94.0
1.00	1127	0.92	92.0
2.00	2280	1.79	89.5
3.00	3419	2.89	96.3
5.00	5622	4.86	97.2

TABLE 2. Comparison of Uranium Recovery by Ethyl Acetate Extraction and Ion-Exchange Separation

Sample number	U_3O_8 added (g.)	Alpha activity by ethyl acetate extraction method (dpm)	Percent recovery	Alpha activity by ion-exchange method (dpm)	Percent recovery
1	$5x10^{-3}$	4280	76.1	5367	95.5
2	$5x10^{-3}$	4443	79.0	5448	96.9
3	$5x10^{-3}$	4239	75.4	5272	93.7
4	$5x10^{-3}$	4396	78.2	5310	94.4
5	$5x10^{-3}$	4472	79.5	5400	96.1
6	$5x10^{-3}$	4372	77.8	5338	94.8

TABLE 3. Recovery of Uranium in Spiked Soil Sample*

Sample taken (g.)	U_3O_8 added (g.)	Alpha activity found (dpm)	Percent recovery
0.2500	$5x10^{-3}$	4400	80.8
0.5000	$5x10^{-3}$	4565	83.8
1.0000	$5x10^{-3}$	4724	86.7

*Analysis of the soil sample for plutonium showed 181.2 pc.

TABLE 4. Recovery of Uranium in Spiked Air Filter Samples

U_3O_8 added (g.)	Alpha activity found (dpm)	Percent recovery
$1x10^{-5}$	7.5	75.0
$5x10^{-5}$	44	84.6
$1x10^{-4}$	100	87.7
$2.5x10^{-4}$	226	87.9

TABLE 5. Recovery of Uranium in Spiked Water Samples

U_3O_8 added (g.)	Alpha activity found (dpm)	Percent recovery
$1x10^{-5}$	8.4	84.0
$5x10^{-5}$	45.2	86.5
$1x10^{-4}$	101	88.6
$2.5x10^{-4}$	225	87.5

TABLE 6. Alpha Activity for Various Concentrations of Uranium Using Ion-Exchange Method of Separation and Electrodeposition on Nickel-Plated Copper Disc

U_3O_8 added (g.)	Alpha activity found (cpm)	Alpha activity found (dpm)
$1x10^{-5}$	4	9.5
$5x10^{-5}$	22	52
$1x10^{-4}$	48	114
$2.5x10^{-4}$	108	257
$5.0x10^{-4}$	228	543
$1x10^{-3}$	468	1114
$2x10^{-3}$	917	2182
$3x10^{-3}$	1425	3392
$5x10^{-3}$	2289	5448

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